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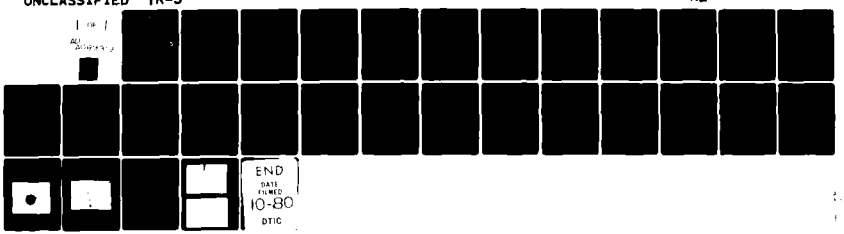

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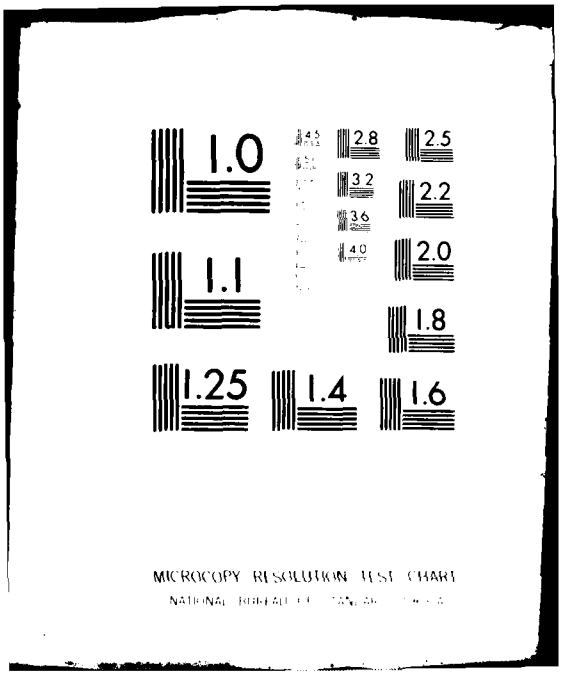
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Direct Digitization System for Quantification in Ion Microscopy

by

B.K. Furman and G.H. Morrison

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A system for the quantification of secondary ion mass spectrometric images produced by an ion microscope is presented. The Microscopic Image Digital Acquisition System (MIDAS) developed in this laboratory by coupling a CAMECA IMS-300 ion microscope, a low light level TV camera, a video color graphics system and digital image processing techniques, is described. Ion images detected by the television camera are converted in real time into matrices of point intensities, maintaining the spatial resolution of the original image. These image matrices may then be converted to ion intensity space, or by the use of standards, to concentration.

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Direct Digitization System for Quantification in Ion Microscopy

B. K. Furman and G.H. Morrison *

Department of Chemistry
Cornell University
Ithaca, New York
14853

ABSTRACT: A system for the quantification of secondary ion mass spectrometric images produced by an ion microscope is presented. The Microscopic Image Digital Acquisition System (MIDAS) developed in this laboratory by coupling a CAMECA IMS -300 ion microscope, a low light level TV camera, a video color graphics system and digital image processing techniques, is described. Ion images detected by the television camera are converted in real time into matrices of point intensities, maintaining the spatial resolution of the original image. These image matrices may then be converted to ion intensity space, or by the use of standards, to concentration. The use of ion implantation to fabricate image standards is presented as an evaluation of MIDAS. Applications of color display as a means of conveying compositional morphological information are demonstrated.

Secondary ion mass spectrometry (SIMS) is a powerful surface analytical technique capable of high elemental sensitivity, small sampling area ($\leq$ 250 μm diameter), and the ability to provide compositional morphological information from solid surfaces (1). Of special interest is the use of ion images to represent elemental distributions at the sample surface. Ion images may be obtained in one of two ways, depending on the instrumentation used. The ion microprobe (2,3), using a primary ion beam of small diameter generates an image from the sum of sequential point analyses with the probe operating in the scanning mode. The principal disadvantages of this method are the time required to generate images with low signal levels and the low resolution of the image. In contrast, the ion microscope (4), a direct imaging SIMS instrument, produces an elemental image of an entire field of view by a method of simultaneous multipoint detection. The resulting image contains information similar to that collected by the scanning technique but is obtained in a fraction of the time with superior resolution.

An ion microscopic analysis produces a large amount of multidimensional information, both spatial and elemental, which can be assimilated only qualitatively by visual interpretation. Quantitative evaluation of ion images requires the ability to evaluate each image at the picture element (pixel) level and to then recombine pixels to yield meaningful information about microstructures within the image field. Previously, images have been recorded photographically, digitized off-line with a microphotodensitometer and then processed by digital image processing techniques (5). A major problem with

this approach is the time required to expose low signal level images on film (> 100 sec) and to process and digitize images (several hours). Also, in generating ion images requiring very long exposures, it is possible for the sample to change considerably due to the sputtering of hundreds or thousands of atomic layers during the acquisition of a single image. In addition, an image which requires an exposure longer than 10 seconds due to a low signal level is not visible on the fluorescent screen. This makes it difficult to discriminate meaningful distributions from noise without long exposures and subsequent lengthy post-exposure processing.

In this paper we present a new system for direct acquisition and digitization of ion images using a low light level silicon intensified target camera coupled with an image intensifier (ISIT). The ion image produced on the fluorescent screen of the ion microscope is viewed by the camera for real-time digital image processing. The system, Microscopic Image Digital Acquisition System (MIDAS), facilitates high speed acquisition of ion images at low light levels, real-time digitization and image processing, and storage in a format compatible with off-line digital image processing routines. The use of color in the display of images, made possible by use of a full color graphics processor, is also presented. Procedures are developed to correct images acquired by the low light level TV camera to ion intensity, and, given standards, to concentration. Ion implantation is used to fabricate "image standards" for evaluating the accuracy of the system.

EXPERIMENTAL

Instrumentation. The hardware system used in these studies is shown schematically in Figure 1. Images were produced by a CAMECA IMS-300 ion microscope which was developed by Castaing and Slodzian (4) and described in detail in the literature (1). Image detection is accomplished by means of a low light level television camera mounted outside the vacuum system of the instrument. At the object focal point of the camera is a fluorescent screen contained within the instrument. Since the final mass-filtered ion beam is projected onto the cathode of an ion-to-electron image converter and the secondary electrons emitted from the converter are accelerated to 20 keV and focused onto the fluorescent screen, as diagrammed in Figure 2, actual detection is of a photon image.

A Quantex QX-26 intensified silicon intensified target (ISIT) camera views the fluorescent screen through an F/.95 50-mm lens with a +4 closeup lens. Optics were designed to allow the 25-mm diameter image on the fluorescent screen to be focused on a 16-mm diameter target within the TV camera at a distance of approximately seven inches. The optics were designed to preserve the spatial resolution of the instrument and to allow maximum transmission of signal to the TV detector.

The camera is capable of recording images over a large range of intensities by means of a variable gain control and an adjustable image intensifier. The resulting gain is sufficiently large that as few as five or six photons will produce a video pulse detectable above the readout noise. Short time exposures can be recorded by turning off the scanning electron beam in the camera while a charge pattern is accumulated on the camera target. When the beam is turned on, the video signal produced can be stored by the

processor to produce a flicker-free display on the monitor.

Typical gain for an SIT tube is about 2000. The image intensifier fiber optically coupled to the SIT camera provides image preamplification of 40 to 50 which increases the overall gain to a maximum of about 80,000. Output of the camera is in the form of a standard RS-170 analog video signal. This output can be directed either to a color monitor for direct viewing or to a digitizer contained within the processor unit.

The processor unit consists of a Grinnell graphics display system with 256 x 256 x 12 bit memory and a 6-bit video digitizer with the ability to digitize, and store a full video frame in real time (1/30 sec). It also allows summation, subtraction, and recursive summation of multiple video frames in real time. In addition to real-time processing, images can be displayed in 256 grey levels, in up to 4096 hues and intensities of pseudo-color, or multiple images (up to three) displayed as 16 intensity levels each in red, green or blue. Pseudo-color is that which is arbitrarily assigned to a given intensity. Image data are stored in solid state refresh memory channels that are periodically scanned to provide a standard video output signal. Refresh memory channels use 4K MOS random access memories to provide rapid access (1.5 μ sec) to any point on the display. Images digitized and processed by the Grinnell unit can be either stored or further processed by transferring them through a bidirectional parallel data transfer interface between the Grinnell and a PDP 11/20 minicomputer.

The computer facilities consist of a PDP 11/20 with 24K words of core memory; a 1.2M-word removable cartridge disk used for the RT-11 operating system and software; a 2.4M-word dual capacity fixed disk assembly and a

9-track magnetic tape unit for potentially unlimited data storage space; an electrostatic printer/plotter for hard copies of program and digitized images; and a GT40 Vector graphics display terminal.

Computer Programs. Applications programs were written primarily in FORTRAN IV, and device handlers were written in MACRO assembly language. Image acquisition and preliminary processing were accomplished by the Grinnell processor. This processor controls most image functions through a set of 26 independent instructions which can be utilized in any sequence. Internal control by the processor with a set of powerful display, acquisition, and generation instructions greatly reduces processing time by eliminating the need for complex MACRO instructions and lengthy FORTRAN routines. The PDP 11/20 transfers instructions and data to the Grinnell processor via a 16-bit bidirectional parallel interface. Image acquisition is controlled by activiting the graphics digitizer within the Grinnell processor.

This option allows one to digitize an image into a 248 x 256 pixel array with 6-bit intensity resolution (0-64), and to store, in real time, a complete frame of this video data in the display system's memory. Digitized data may replace data already stored in the memory, may be directly added or subtracted to data already stored in memory, or may be recursively added to data already stored in the memory. In all of these modes the operator may decide to digitize and store a single frame of data, a specified number of frames of data (up to 255), or to operate continuously until the host computer halts operation. After an acceptable image is acquired it can then be transferred

to disk and processed by the PDP 11/20 using the existing digital image processing software (6). A variety of software for the manipulation and display of digitized images through the Grinnell processor have also been developed, including mathematical, enhancement, display, and transfer routines. These subprograms represent a basic set of useful routines for acquisition, manipulation, and display of ion images either in real time or for off-line processing. All routines are contained within the general program IMAGE, which consists of a keyboard command interpreter that supervises calls to the FORTRAN or MACRO subroutines. The structure of IMAGE is such that expansion and change is readily facilitated.

Details concerning the operation and structure of IMAGE or the system are available upon request from the authors.

Sample Preparation. Standard image files were obtained using a highly polished aluminum disk. The $^{27}Al^{+}$ signal was used for calibration of fairly high signal levels, whereas low signals were obtained using several molecular ion peaks produced by the sample. Signal levels ranging from less than 100 cps to greater than 10^{6} cps were produced by varying the primary ion current and the detected mass peak. Instrumental operating conditions are given in Table I.

Image Standards. An Accelerators, Inc. model 300R ion implanter was used for the implantation work. In the ion implantation process (7), an ion beam of almost any element or isotope can be implanted into the near surface region of a sample. The peak of the implanted distribution depends on the incident energy, and the peak concentration depends on the implant fluence. The ion beam is rastered to ensure uniformity of doping in the lateral directions for an area as large as 5 cm x 5 cm.

In fabricating the image standards the sample was covered by a metal mask comprised of horizontal slits 20 micrometers wide spaced 150 micrometers apart. This was placed over a sample of highly polished Si(111) and implanted with ^{115}In to a fluence of 1.8×10^{15} atoms/cm^2 at an energy of 250 keV. The mask was then rotated 90° and implanted with a ^{115}In fluence of 5.0×10^{15} atoms/cm^2. The resultant implant was a pattern of crossed stripes of three concentrations plus background. Samples were analyzed by SIMS under standard operating conditions listed in Table I.

RESULTS AND DISCUSSION

The pivotal step in quantification of the ion image is the determination of the relationship between the digitized intensity of the TV camera and the ion intensity recorded by the photomultiplier detection system of the ion microscope. Ion intensity is defined as the integrated signal obtained on the photomultiplier detection system. This assumes one ion produces one electron which in turn produces one photon to be detected. Rudat and Morrison (10,11) have shown some detector discrimination in the ion-to-electron conversion which must be considered to report true ion intensity. Since the effect is small and does not affect results obtained using standards it was ignored for the described studies.

Previous studies (5,6,8,9) with photographic recording and subsequent development and digitization accomplished this step through determination of the characteristic curve of the film and the conversion of film darkening per pixel to ions extracted per pixel. By this method a new characteristic curve must be determined for each new roll of film and operating conditions must be precisely reproduced. In the direct digitization system

described here a characteristic curve of the TV detection system response must also be determined. This calibration can be done once and is not affected by changes in film, development conditions or digitization conditions. MIDAS is calibrated by recording a series of standard images of varying intensities from background to saturation at different gain settings of the camera. These standard images define the curve of recorded intensity vs ion intensity. The intensity of image points of experimental images are then calculated from the mathematical representation of this calibration.

The standard images are made of an aluminum disk in which the field of view is limited by a 150-μm diameter aperture to overlap the 125-μm diameter field of view of the photomultipler. The output of the photomultiplier is an integration over the entire area of the detector. For a conversion from recorded intensity to ion intensity for each pixel one must calculate the average counts/sec/pixel at the photomultiplier and relate it to the intensity of each pixel recorded by the camera. To do this one must assume every point on the photomultiplier has the same efficiency for detecting electrons. It is known that there is a slight loss of light collection efficiency around the edges due to the optics that carry the light to the photomultiplier, but this area was considered negligible and was not introduced into our calculations. Figure 3 represents the recorded image of the standard image file.

With only 6 bits of digitization (64 levels) occurring for each pixel in the image, only a small range of intensities can be recorded. To alleviate this problem the gain on the ISIT camera can be varied. Images can then be recorded over a range of four orders of magnitude. An analog-to-digital converter monitors the gain at which an image is acquired and records it for each

image. To calibrate the system, a series of standard images of varying intensities were recorded at a constant gain setting. This procedure was repeated over the entire range of gain settings, and resulted in a set of parallel lines with the y-intercept a function of the camera gain when the log ion intensity (I) was plotted as a function of recorded pixel intensity. The log values of the y-intercepts were then plotted as a function of the log of camera gain. Combination of these two plots yielded an equation which describes the ion intensity over the entire area of the photomultiplier as a function of the log of camera gain (G) and average pixel value ($\bar{p}$) of that area. Ion intensity per pixel (I_p) may then be obtained by dividing by the area of the photomultiplier, A_p, as represented by Equation 1.

$$\log I_p = \frac{0.016(\bar{p}) - 4.1(\log G) + 20.8}{10^{A_p}} \qquad (1)$$

Another feature of the system is the use of a fiber optic image intensifier coupled to the SIT camera tube. Three options are available in using this mode: 2x, 4x, and 10x intensifier gain. Standard images are the same as described earlier. Due to the large amount of noise present with this mode it was necessary to average up to 255 frames to obtain reasonable images. Quantification of images using one of these modes is accomplished using Equation 1 where $\bar{p}$ represents the recorded pixel intensity divided by the calibrated intensifier gains 2.1 , 4.1 and 9.2 depending on the intensifier gain 2x, 4x or 10x.

For still lower light levels the feature of integration by summing multiple frames can be used. Quantification is accomplished by using Equation 1 but where $\bar{p}$ represents the summed pixel value divided by the number of frames summed. In quantifying images obtained from summing multiple frames, care must be taken to avoid saturation of the image, which can result in loss of information by overflow of memory. Because of the 6-bit digitization and 12 bits of memory available, the most intense signal can be summed for only 64 frames ($\sim$ 2 sec) without saturation occurring. For the least intense signal (value of 1 on a scale of 0-64), 4095 frames ($\sim$ 135 sec) can be added. For signal levels this low the technique of photon counting (12) would be preferential to summation because of an enhanced signal-to-noise ratio.

Sensitivity. One major advantage of MIDAS is its enhanced sensitivity as compared to visual observation or photographic detection. Table II represents a comparison of the three techniques for detection of ion images. Advantages of MIDAS over visual observation are increased dynamic range and the ability to quantify the image. MIDAS advantages over the photographic detection system include increased dynamic range, increased sensitivity, and reduced acquisition time. The latter not only involves the amount of time needed to acquire adequate signal but also the time required to process, digitize and calibrate the film if quantitative information is desired. The major disadvantages of MIDAS are the decreased quality of the image at high signal levels, cost, and noise at very low light levels.

Resolution. In designing the optics system for MIDAS two goals were considered important; first, to provide for maximum sensitivity by using a high speed lens system, and second, to preserve the 1-μm spatial resolution of the instrument. The system was also limited by the physical characteristics of the instrument, i.e. focusing very close (150-mm object distance) on a 25-mm screen (object size) and completely filling a 16-mm target (image size) in the television camera. Using Equation 2 an optimum focal length of 100 mm was calculated.

$$\frac{\text{object size}}{\text{object distance}} = \frac{\text{image size}}{\text{focal length}} \qquad (2)$$

Since no commerical c-mount 100-mm lenses were available, a 50-mm F/.95 lens with a 2x extender was selected. To allow the lens to focus at a distance of 150 mm, a +4 closeup lens was used. To allow greater sensitivity when resolution is less important, the 2x extender is omitted, which results in a smaller image but lower light detection limits. Because of the different speeds and magnifications of each lens system, image quantification is also dependent on lens selection. Quantitative results presented in this paper were all obtained using the 50-mm lens with no extender.

Multiple Images. Another important feature of the MIDAS display system is its ability to display several images simultaneously by the use of multiple colors. This is advantageous when comparing ion image features of the same sample obtained for different elements or isotopes. One problem that arises is the registration or correlation of the respective image fields. This problem, as encountered in the off-line digitization system, has been studied by Fassett and Morrison (8). With MIDAS a further registration problem is caused due to the shift of the image with mass due to the effect of changing magnetic

fields on the TV camera. Since no rotational movement has been observed, samples are registered by simultaneous display and then a real-time translational (x-y) movement of one image at a time by computer software. This moves the images displayed on the monitor. Results of this are shown in Figure 4 which depicts three images of a semiconductor device.

Image Standards. A major limitation of SIMS analysis is the uncertainty of quantitative information. Variability of the secondary ion yield due to surface states, matrix effects and sputtering effects (13,14) is the principle cause of this problem. Further difficulties in quantitative ion microscopic analysis are caused by crystallographic, topographic and chromatic contrast (15). Thus, the conversion of a compositional micrograph in ion intensity space to concentration space is non-trival.

Previous attempts to quantify non-imaging SIMS data have been pursued by one of two general methods: the application of theoretical ionization models such as the Local Thermal Equilibrium (LTE) model originated by Andersen and Hinthorne (16), or empirical methods such as the use of relative sensitivity factors (17). Several attempts to quantify ion images using theoretical models have been reported using LTE correction (18), sensitivity factor correction (17), total current monitoring (19) and application of simple correction factors (20).

The use of standards for quantifying ion images was first presented by Fassett, et al. (5) using Ni-Cu foils. Drummer and Morrison (21) recently used "imaging standards" similar to those presented in this study as a means of evaluating the theoretical and empirical calibration approaches. The application of standards for quantifying images has shown itself to be a superior method of quantification. The problem that arises is that of finding standard

materials of both known concentration and homogeneity of trace element composition (22). To alleviate this problem the technique of ion implantation was employed to fabricate image standards for use in the generation of a working curve for the conversion of ion intensity to concentration. Using striped implants as described earlier, three points of known concentration can be extracted from each image acquired. By recording images at various points throughout the depth profile of the implants, a series of values ranging over several orders of magnitude can be obtained. In Figure 5 the depth profiles at each of the three fluences (concentrations) are shown. Images were recorded at ∿ 50-sec intervals from the surface (minimum concentration) to the peak of the implant (maximum concentration). Recorded intensities from the images are then converted to ion intensities as described earlier. Ion intensities may then be converted to concentration by the method previously reported by Leta and Morrison (23). Results and an error analysis are presented in Table III.

After this calibration curve of ion intensity vs concentration, which is linear over the range of the implant, has been calculated, it can be applied as a correction to any ion image previously recorded. Figure 6a represents the ^{115}In image as recorded by the MIDAS at peak concentration. This image may then be converted to ion intensity by Equation 1. A final conversion of the image to concentration is shown in Figure 6b. One problem inherent with this system which can be seen in the preceding images is that of camera "blooming", the spread of signal from areas of high intensity into adjacent pixels. The problem can be reduced by recording images at lower camera gain to reduce the overflow of signal to adjacent areas within the camera target.

The use of image standards can be applied to any sample in which ion implantation is possible. It can be used as a means to internally dope a material with a specific trace element in known distribution. It can also be used as a standard addition method (24) to quantify information from ion images.

CONCLUSIONS

The MIDAS System provides a convenient way to quantify images in ion microscopy, a multidimensional technique producing simultaneous three-dimensional spatial and elemental information about a sample. The combined use of ion imaging, MIDAS, and a digital computer simplifies image acquisition and the complexities of handling the large amount of data involved in extracting this multidimensional information. The rapid image acquisition and digitization, and the ability to access individually any point or points within the field of view allows the obtaining of depth profiles of several areas of the sample. Areas profiled may range from a 1 pixel x 1 pixel area up to a full digitized field (256 x 256 pixels, 250-μm diameter), thereby providing multielement multiarea depth profiling in real time. The use of pseudo-color for display of images facilitates the transmission of maximum information to the viewer.

Other possibilities for quantification of images, such as matrix ratioing and the use of theoretical and empirical calibration corrections on whole images in real time, will be investigated. Additional aspects of digital image processing such as edge enhancement, feature isolation, and pattern recognition will be studied using the MIDAS system. Since the goal of ion microscopy is

the chemical analysis of unknown microstructures, the use of the MIDAS system to acquire, manipulate, correct, and redisplay images may soon allow the display of accurate chemical composition in near real time. Finally, the capabilities of this novel system should be applicable to other imaging microscopic analytical techniques.

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CREDIT

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Table I. Instrumental Parameters

Instrument: CAMECA IMS-300 ion microanalyzer

Primary ion beam species: O_2^+

Primary ion current: 10 nA - 5 mA

Instrument pressure: 2×10^{-7} torr

Mass resolution: 300

Detection system: photomultiplier pulse counting, ion imaging

	Positive Secondary Ions	Negative Secondary Ions
Crater	700 x 700 μm	580 x 580 μm
Sampling area:	35 - 225 μm	35 - 190 μm
Primary ion energy:	5.5 keV	14.5 keV
Incidence angle:	33° from surface	57° from surface

Table II. Sensitivity Comparison

Photomultiplier Current (A)	Visually Detected	Photographic Exposure (sec)	MIDAS Detection: Gain (Arbitrary Units)	MIDAS Detection: Frames (1/30 sec)
5×10^{-12}	Yes	1/50	2,500	1
1×10^{-13}	Yes	1/10	4,500	1
1×10^{-14}	Yes	1	5,500	1
1×10^{-15}	No	10	8,000	1
5×10^{-16}	No	50	11,000	1
1×10^{-16}	No	100	17,250	1
5×10^{-17}	No	> 500	17,250	30
1×10^{-17}	No	>> 500	17,250	> 255*

* Photon counting for several minutes

Table III. Analysis Results and Error Analysis

Fluence, atom/cm^2 x 10^{-15}	MIDAS Measurements: Recorded Intensity,[a] counts per pixel	MIDAS Measurements: Concentration, atom/cm^3 x 10^{-20}	Photomultiplier Measurements: Ion Intensity, cps	Photomultiplier Measurements: Peak Concentration,[b] atom/cm^3 x 10^{-20}	Relative Error, %
1.8	20	1.9	38,300	1.6	18.8
5.0	42	4.2	85,000	5.2	19.2
6.8	54	6.0	138,000	6.7	10.4

[a] Gain = 10,800, recorded at peak implant (maximum concentration)

[b] Average $\bar{n}$ of three profiles using the photomultiplier system under identical conditions

CAPTIONS

Figure 1. Block diagram of system layout.

Figure 2. Image detection schematic illustrating both the TV image acquisition system and the photomultiplier detection system used for calibrating MIDAS.

Figure 3. Color display of ^{27}Al standard image file. Color scale represents recorded intensity in counts/pixel (CP). Field of view: 150 μm diameter.

Figure 4. Composite color display of Al, Si, and Na (impurity) ion intensity distributions in a semiconductor device. Field of view: 200 μm diameter.

Figure 5. Depth profiles of ion implanted image standard. Silicon sample implanted with ^{115}In at a fluence of (1) 1.8×10^{15}, (2) 5.0×10^{15}, and (3) 6.8×10^{15} atoms/cm^2.

Figure 6. Color display of ^{115}In distribution in ion implanted image standard (Si < 111 >). (a) Recorded intensity, counts/pixel (CP), 0-64 levels; (b) concentration, ppm, 100-1000. Field of view: 200 μm diameter.

Fig 1

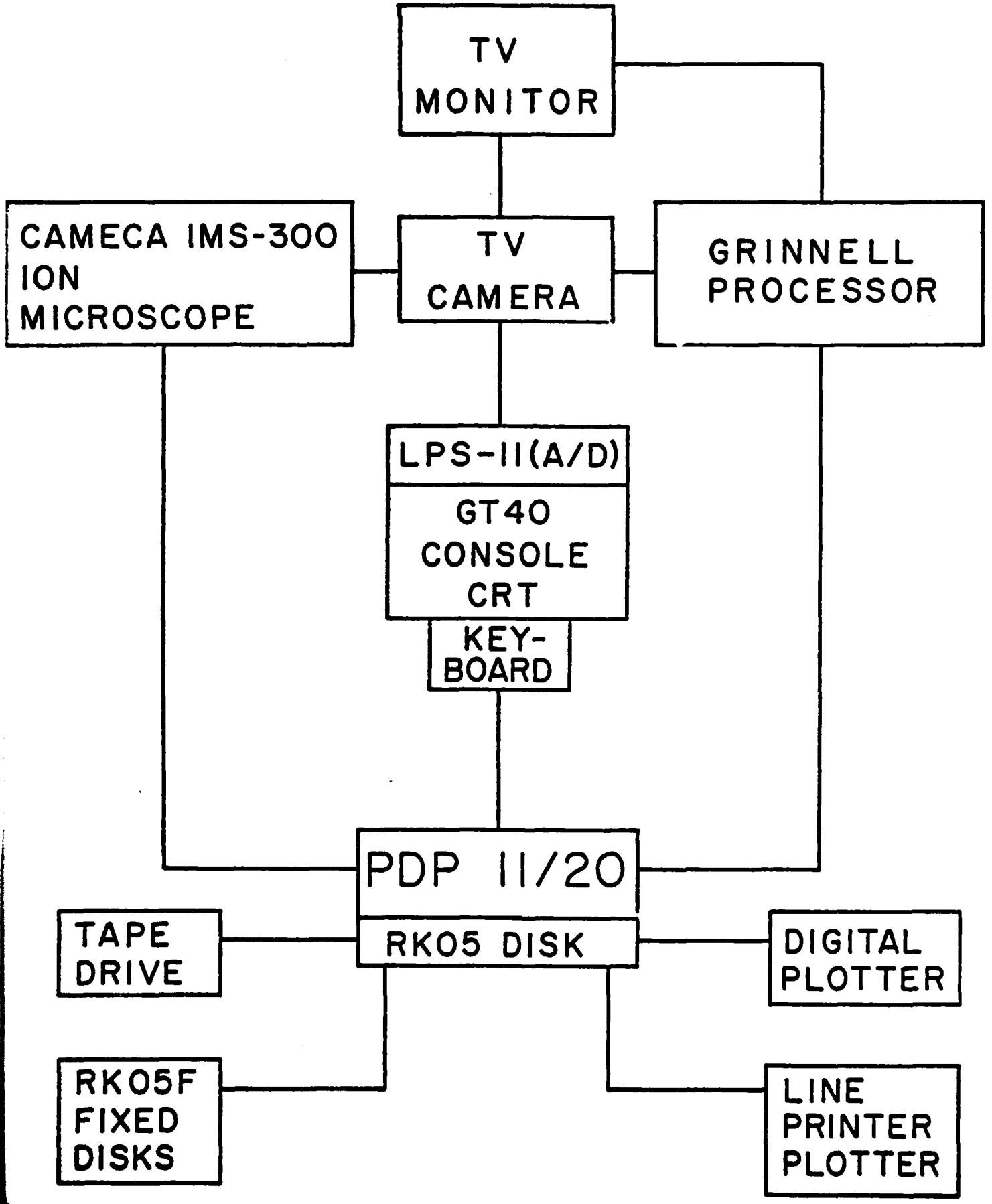

TV MONITOR
CAMECA IMS-300 ION MICROSCOPE
TV CAMERA
GRINNELL PROCESSOR
LPS-11(A/D)
GT40 CONSOLE CRT
KEY-BOARD
PDP 11/20
TAPE DRIVE
RK05 DISK
DIGITAL PLOTTER
RK05F FIXED DISKS
LINE PRINTER PLOTTER

Fig 2

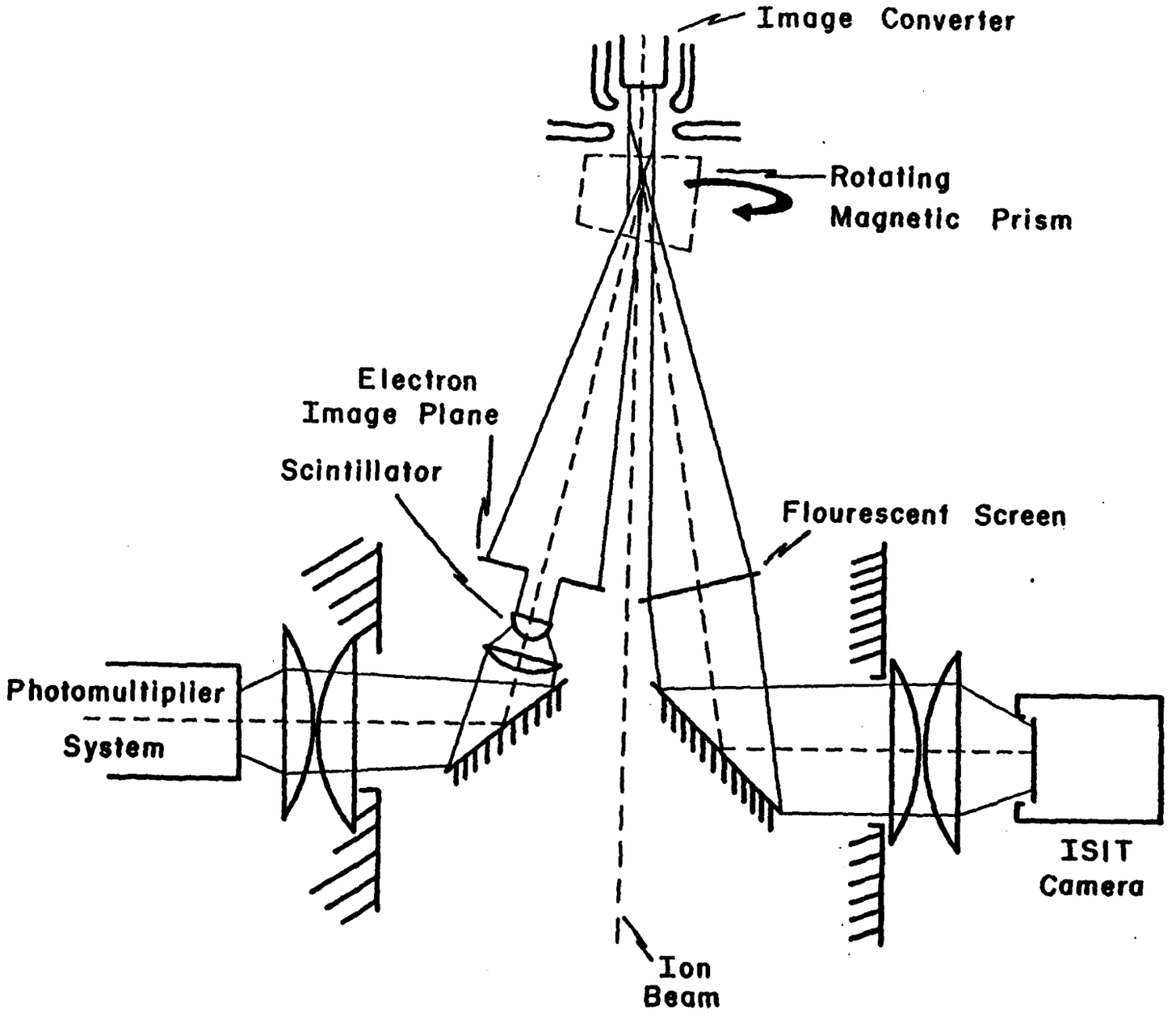


Image Converter
Rotating
Magnetic Prism
Electron
Image Plane
Scintillator
Flourescent Screen
Photomultiplier
System
ISIT
Camera
Ion
Beam

Fig 3

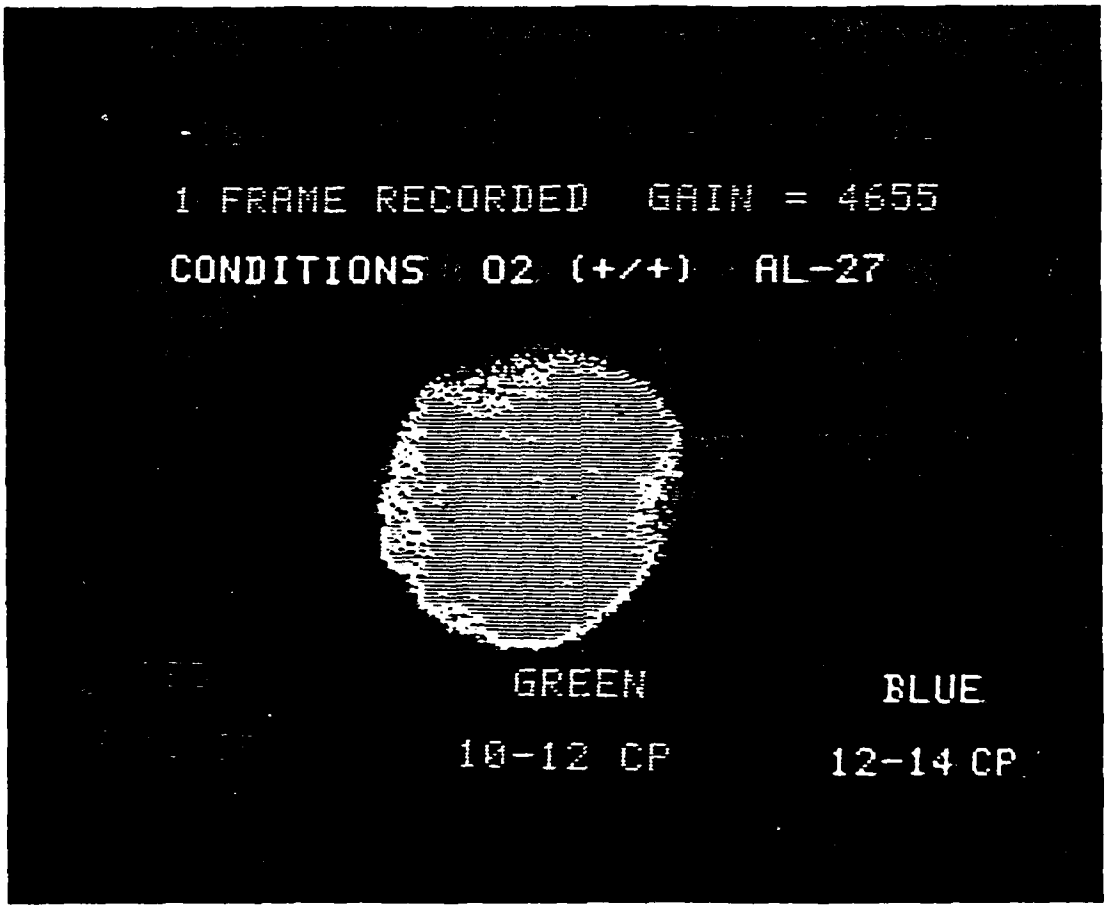

1 FRAME RECORDED GAIN = 4655
CONDITIONS O2 (+/+) AL-27
GREEN
10-12 CP
BLUE
12-14 CP

Fig 4

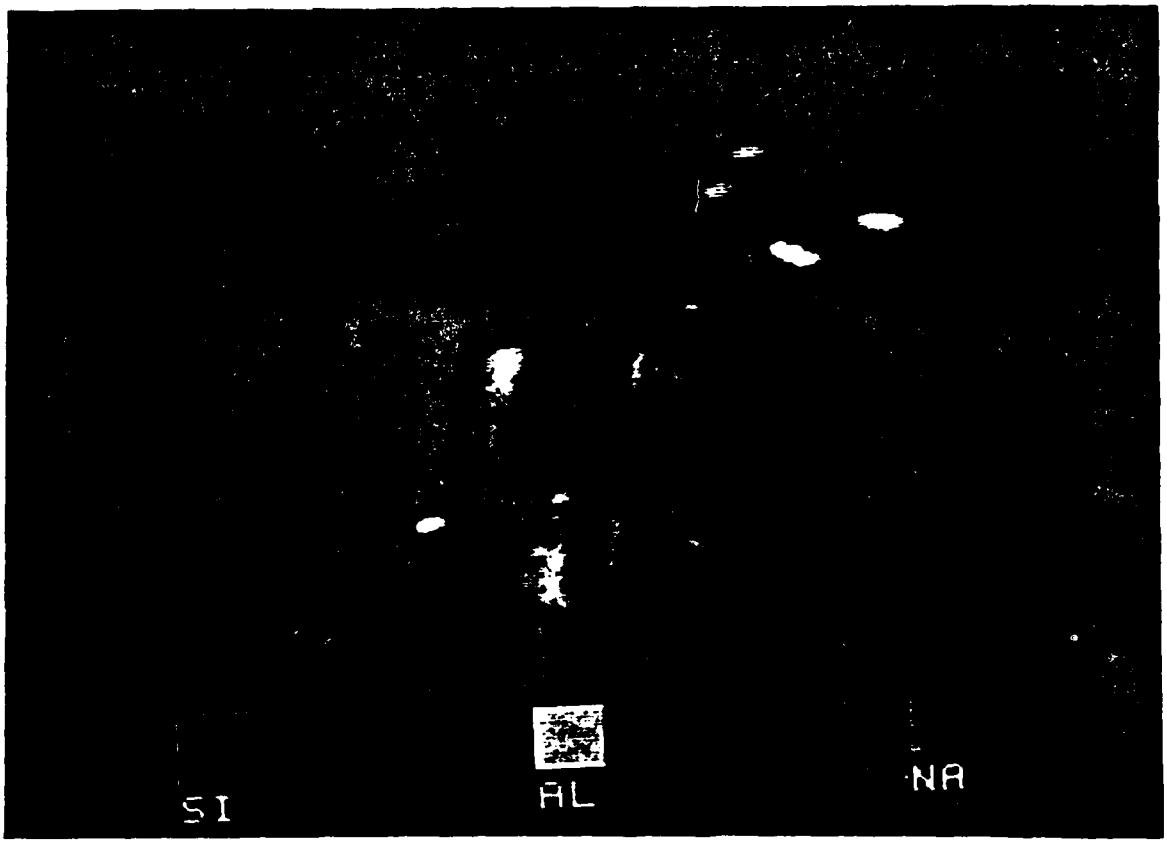


Fig 5

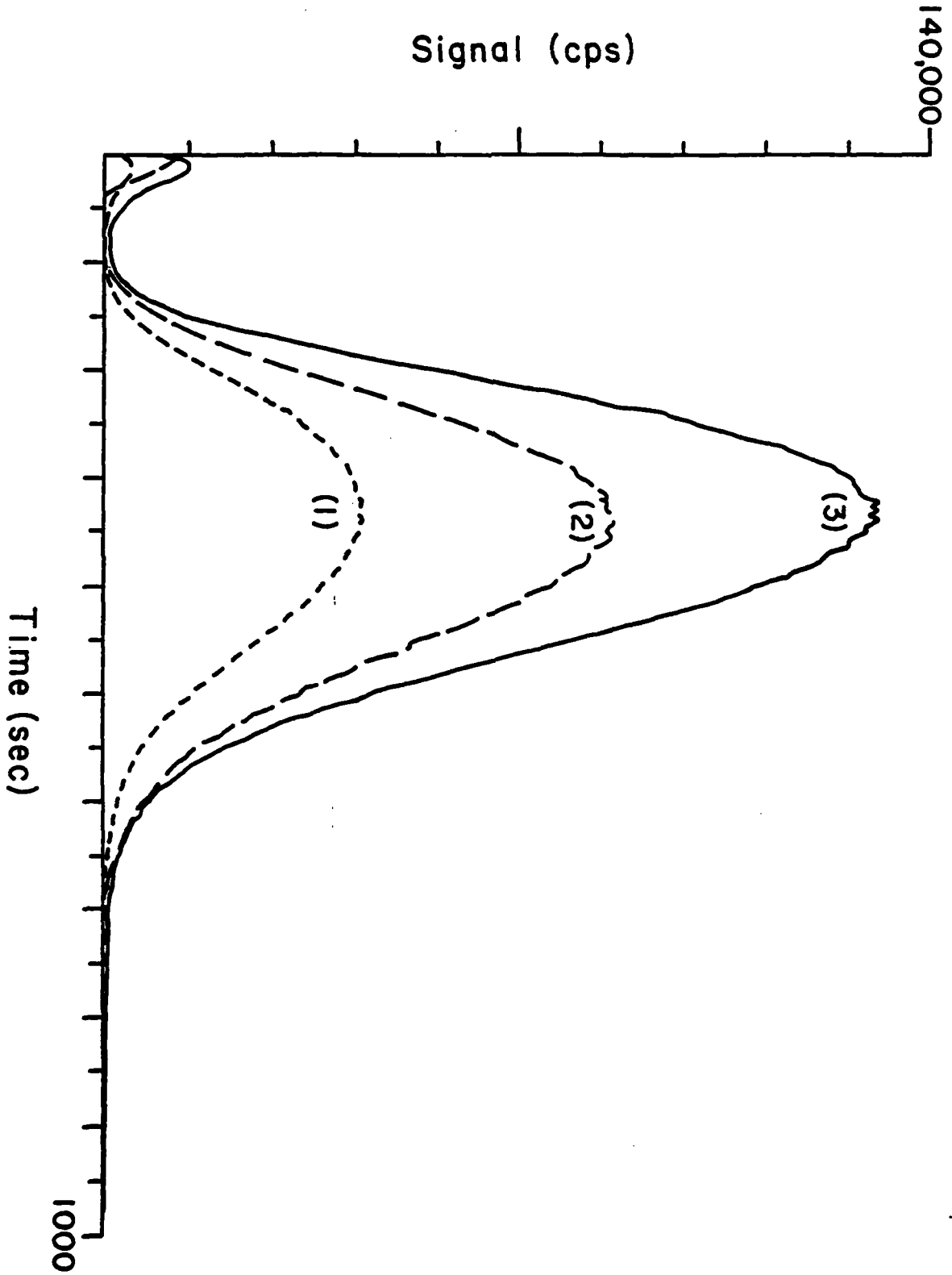
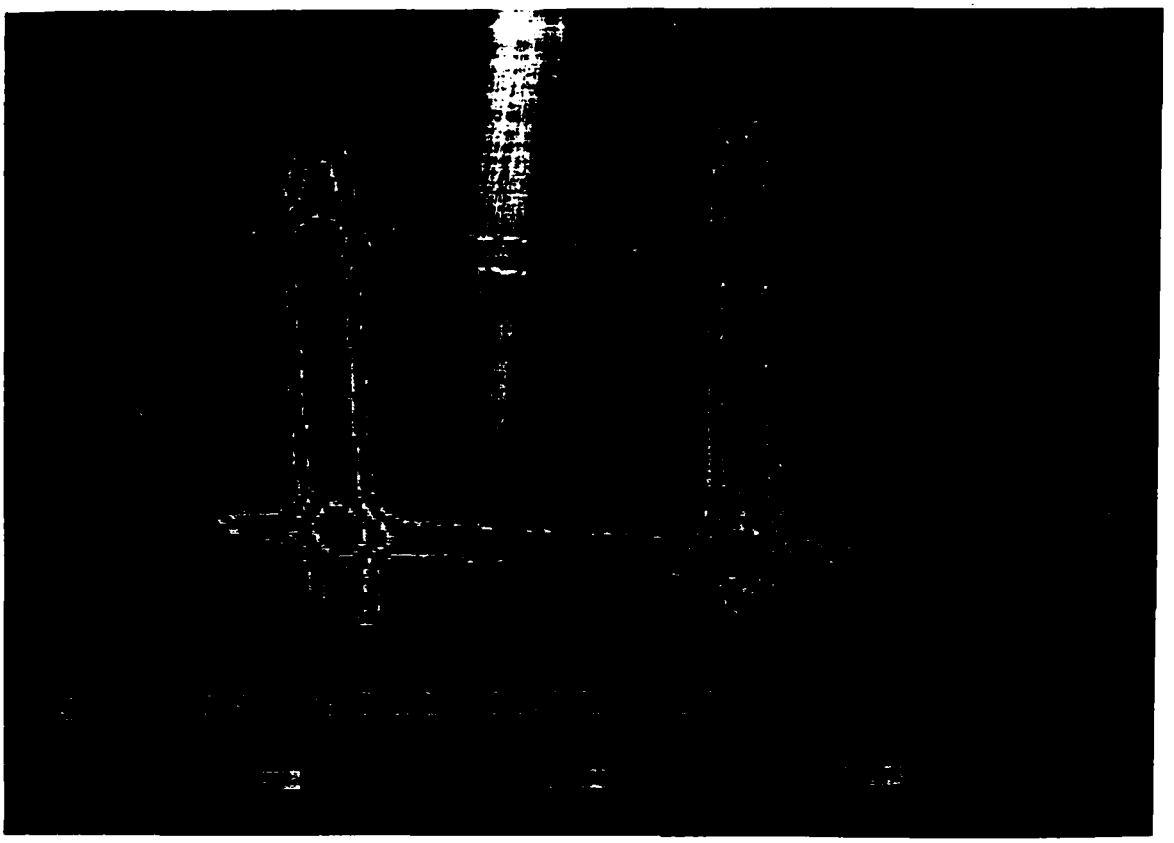
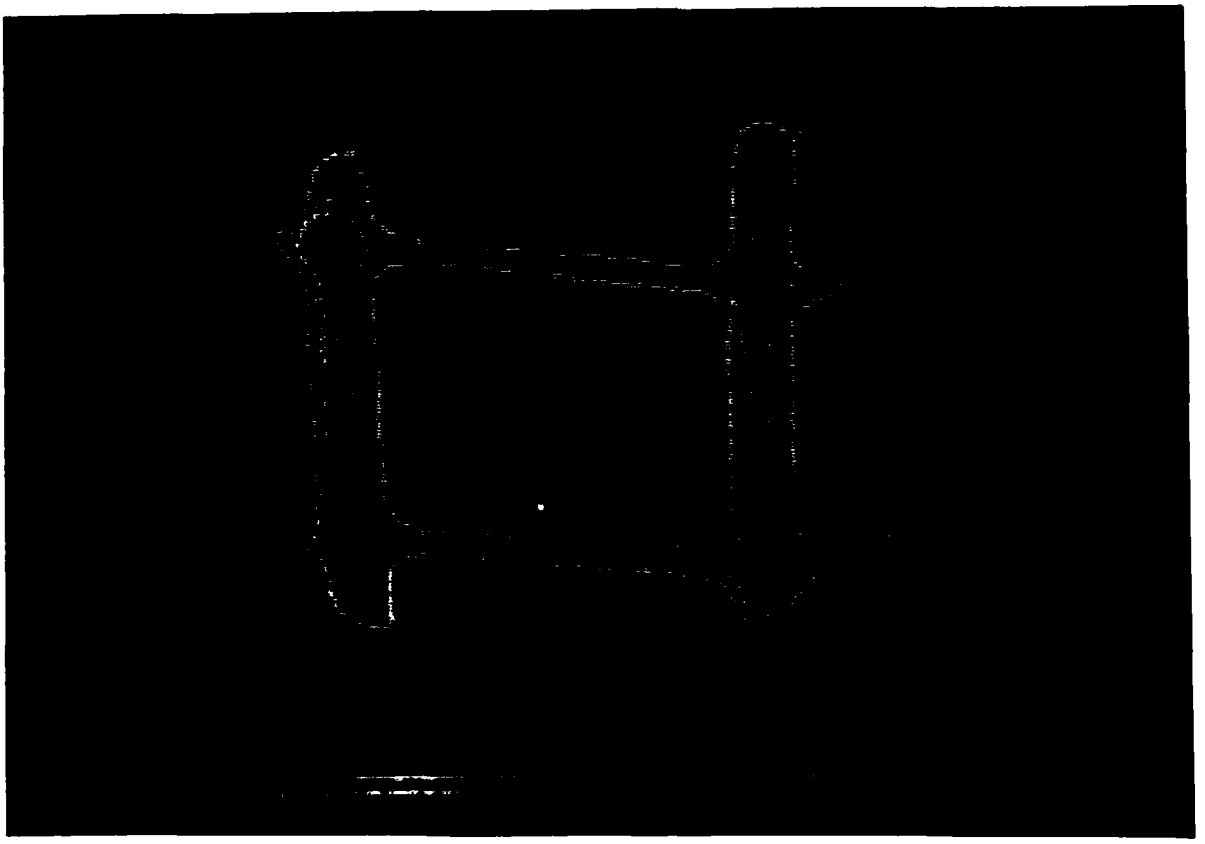


Fig 6.

a)



b)



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